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VISCOSITY EFFECT ON FLOW ORIENTATION OF SHORT FIBERS

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Monsanto Research Corporation

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When resin viscosity is lower than 800 poises, plug flow is observed in 30 to 50 vol. % suspensions flowing through uniform and convergent channels. The alignment of fibers in the flow direction occurs exclusively in convergent channels. When resin viscosity is higher than 1,000 poises, velocity profiles observed in uniform and convergent channels are similar to those for Newtonian fluids but blunted near the channel center. The alignment of fibers occurs in both convergent and uniform channels. The fiber alignment achieved through a convergent channel flow increases as resin viscosity increases. Resin - fiber separation decreases with increasing resin viscosity. The flow resistance of 40 vol. % suspensions extruded through a 60 degree convergent channel slightly increases with an increase in resin viscosity up to 1,000 poises and sharply increases above this resin viscosity.

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FOREWORD

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INTRODUCTION

Fabrication of short fiber/thermoset composites poses many rheological problems, being compared to the continuous filament composites, particularly when the fibers are short and their orientation is to be controlled by a flow process. The property level of short fiber composites is limited by several parameters,[1-3,8] such as fiber orientation, distribution and breakage, void content and the adhesion of resin to fiber surfaces, as well as by other factors such as fiber aspect ratio (length/diameter), modulus, strength and concentration, in addition to matrix properties. Control of the above parameters and variables during a flow process is important if one is to utilize the superior properties of reinforcing fibers, and also to provide reproducibility and reliability of the fabricated composite.

Conventional molding techniques developed for thermoplastics, such as transfer-molding, injection molding and extrusion, are often used to fabricate composite materials with thermoset matrices.[3-8] However, several difficulties arise with flow processings of short fiber composites under the operating conditions established for homogeneous thermoplastics. At

high fiber concentrations, predominant fiber - fiber interactions result in a plug flow in uniform channels and unsteady flow in convergent channels.[9] When the fiber length is comparable to the dimensions of flow channels, both translational and rotational motions of fibers are restricted by the channel wall. Fiber orientation is primarily determined by the local velocity gradients around the fiber center.

One way of establishing optimum conditions for a flow process of short fiber composites is a direct observation of fiber behavior in concentrated suspensions as a function of several operating variables.[9-11] In a previous paper,[9] the effects of fiber concentration, die-geometry and flow rate on the fiber behavior and flow properties of concentrated suspensions were reported. The viscosity of resin used in the experimental study was as low as 55 poises, which is approximately equal to the resin viscosity (44 poises) observed[10] for a typical epoxy resin with a short thermal history at 125°C.

The behavior of fibers suspended in concentrated suspensions is determined by the hydrodynamic and mechanical interactions of the fiber with surrounding fibers, resin and nearest wall.[11] The drag force due to hydrodynamic interactions is a function of the resin viscosity and relative movement of fibers to surrounding resin. In thermoset matrices, the resin viscosity varies from 1 to 10,000 poises or higher, as the resin reacts with hardener, before the resin stops flowing.

Short fibers easily form clumps and separate from the low viscosity suspending medium.[9] As the resin viscosity increases, however, fibers are dispersed to a great extent and there is less separation of fibers from the resin.

It is the purpose of this paper to report experimental results and discuss the effect of resin viscosity on the short fiber orientation, distribution and deformation in concentrated suspensions, and on the flow properties of the suspensions. The viscosity of resins is varied from 15 to 25,600 poises. Rectangular channels are used to eliminate the optical distortion caused by channel walls.

EXPERIMENTS

Suspensions: Behavior of short fibers is cinematographically studied in flowing suspensions with high fiber concentrations, using a technique developed by Mason and his coworkers.[11] To observe the behavior of tracer fibers in concentrated suspensions with 5 to 60 vol. % fibers, optically distortion-free transparent suspensions are prepared from 1/8" long, heat-treated, E-glass fibers and various resin mixtures. Resin mixtures used in this study are i) Epon Resin 828 (Shell Chem. Co.) and n-butylamine polymerized in the presence of n-butyl phthalate and ii) Epon Resin 828 and aniline polymerized in the presence of n-butyl phthalate. The viscosity of resin mixtures is varied from 15 to 25,600 poises while the refractive

index is matched to that of E-glass fibers. The tracer fiber also consists of 1/8" long, heat-treated, E-glass fibers, but colored with chromium oxides. The tracer fibers were mixed, with the ratio of 1:200, to other colorless fibers. Fibers are mixed with resin mixtures prior to flow experiments, as described in a previous paper.[9]

Measurements: To determine the mechanism of fiber orientation, distribution and deformation during flow processes, the velocity profile and the orientation of tracer fibers located in the central plane of flow channels are quantitatively analyzed. Effects of resin viscosity, flow rate, fiber concentration and die-geometry on fiber distribution and orientation are quantitatively studied. Deformation of tracer fibers and migration of visible voids are qualitatively studied. Flow properties of concentrated suspensions are also measured using an Instron tester. All flow experiments are carried out in uniform and convergent rectangular channels at 24°C ± 0.5°C. The thickness of the channels is 1/8" in all cases. The width of flow passage is decreased from 1" (top) to 1/4" (bottom). The total angle of convergent channels which connect the top to the bottom uniform channels is varied from 20° to 210°. The length of the bottom channel is 1.0".

RESULTS AND DISCUSSION

In concentrated fiber suspensions, both translational and rotational motions of a specific fiber are affected by the interaction of the fiber with surrounding fibers, resin and wall. The lag of fibers behind resin and the existence of stagnation region caused by these interactions result in resin - fiber separation and nonuniform distribution of fibers in the suspension. When fibers are in loose bundles, debundling of the fibers is essential to achieve the statistical distribution of fibers in the suspension. The velocity profile in flowing suspensions determines the change in fiber orientation distribution during a flow process. The interaction of fibers with surrounding fibers, resin and wall is a function of the resin viscosity and the relative motion of the fiber to the surrounding.

General trends of viscosity effects observed in the present experimental study are summarized as follows: a) When resin viscosity is low, plug flow is observed in both uniform and convergent channels.[9] Alignment of fibers in the flow direction occurs exclusively in convergent channels. As resin viscosity increases beyond a critical value, e.g. 800 poises for 40 vol. % suspensions of 1/8" long glass fibers, however, velocity profiles of suspensions become similar to those for Newtonian fluids but blunted near the channel center. Fiber orientation distribution changes in both convergent and uniform channels in these suspensions. b) As resin viscosity increases, the

lag of fibers behind resin decreases. The blockade[9] of a narrow exit of convergent channels with fibers decreases with an increase in resin viscosity. c) In convergent channels, fibers in bundles are debundled with increased resin viscosity, as well as with increased flow rate and the decreased relative dimension of the channel exit to the fiber length. The debundling of fibers primarily occurs near the exit of convergent channels, where the highest shear and elongation forces are available. d) As resin viscosity increases beyond a critical value at which the velocity profile of suspensions varies from a plug flow to a blunted, Newtonian type flow, more fiber buckling is observed, particularly at high flow rates. Fiber buckling is observed in 50 and 60 vol. % suspensions independent of resin viscosity. e) As resin viscosity increases, more air bubbles are trapped in dilute suspensions such as 5 to 20 vol. % of fibers. When fiber concentration is increased, the size of trapped air bubbles decreases. However, the air bubbles not only move faster than fibers in flowing suspensions but also coalesce and migrate inwards in convergent channels and outwards in divergent channels.

The effects of resin viscosity on velocity profiles of fibers, resin - fiber separation, stagnation region, fiber alignment through convergent flows and flow properties of concentrated suspensions are quantitatively studied.

i) Velocity profiles of suspended fibers: In homogeneous systems the streamlines in uniform channels are parallel to the surfaces of the channel wall, independent of resin viscosity, whereas in convergent channels the streamlines follow a straight line passing through the vertex of the channel and the fiber center. However, when resin viscosity is low, the paths of tracer fibers in convergent channels often deviate from the straight line passing through the fiber center and the vertex of the channel,[9] and the extent of deviation depends on the interaction with surrounding fibers. The deviation in streamlines is not as great, and the fibers are more uniformly aligned in the flow direction when the viscosity of the resin is high. When the velocity component, V_x, in the axial direction is considered alone, a plug flow is observed in low viscosity resins at fiber concentrations of 5 to 60 vol. % despite the radial component of velocity, V_ψ, observed in the deviation of streamlines. In a medium viscosity resin, such as 444 poises, a plug flow is observed only for 30 to 50 vol. % suspensions. At lower and higher fiber concentrations, velocity profiles are similar to those for Newtonian fluids but blunted near the channel center. The effect of resin viscosity on velocity profiles of fibers was determined in 40 vol. % suspensions. Figure 1 shows the changes in velocity profiles observed in a 1/8" thick and 1.0" wide uniform channel at two different flow rates. For both uniform and convergent channels,

a plug flow is observed when resin viscosity is lower than 800 poises, at low flow rates such as 0.25 in.^{3}/min. However, the fibers located near the channel wall tend to move more slowly than those near the channel center, at high flow rates and in narrow channels, due to the drag force induced by the nearest wall. When resin viscosity is greater than 1,000 poises, velocity profiles become similar to those for Newtonian fluids but blunted near the channel center. The velocity of fibers observed near the channel center is 70 to 80% of that for Newtonian fluids, and 98 to 112% of the mean velocity for homogeneous systems. This indicates the possibility of resin - fiber separation even in high viscosity resins.

ii) Resin - fiber separation: When a plug flow is observed in suspensions, as demonstrated in Figure 1, the ratio of fiber velocity to mean velocity, v_{fiber}/V_{mean}, can be used as a measure of resin - fiber separation. Fiber separation increases proportionally to $(1 - V_{fiber}/V_{mean})$. When the velocity ratio, V_{fiber}/V_{mean}, is unity, there is not lag of fibers behind resin. This fiber separation is due to wall effects[11] on the translational motion of fibers, which become significant even in dilute suspensions when the fibers are located within a fiber length of the wall. Since the length of fibers is comparable to the thickness of flow channels under the experimental conditions used here, the wall effect cannot be ignored in any suspensions. Figure 2 and Tables I and II show the resin - fiber separation observed in 40 vol. % suspensions subjected to flow

through uniform and convergent channels. The lag of fibers behind resin decreases with an increase in resin viscosity and increases with decreasing the sizes of flow channels. More resin - fiber separation is observed in convergent channels compared to uniform channels. In general, this fiber separation decreases with increasing flow rate and increases as the convergence angle increases at high flow rates. A minimum in resin - fiber separation is observed in 40 vol. % suspensions.

iii) Stagnation region: A stagnation region is observed in convergent channels when the convergence angle is larger than 120 degrees in both low and medium viscosity resins. Figure 3 illustrates the relation between the actual convergence angle of the channel and the converging angle of flowing suspensions, observed in a medium viscosity resin (444 poises). The converging angle of flowing suspensions was approximated by projecting motion pictures of flowing suspensions onto a screen at slow speeds. With this condition, the fibers located near the channel wall outside the converging angle do not move, or they move with a very slow velocity which cannot be measured. The stagnation region increases with an increase in resin viscosity at low flow rates such as 0.25 in.^{3}/min., but does not vary when the flow rate is 0.625 in.^{3}/min. It is noted that the angle of converging flow is much smaller than the actual convergence angle of the channel when the latter is as

large as 180 to 210 degrees, and further that fiber suspensions are subjected to shear flow at the boundary of the stagnation region. The existence of a stagnation region results in a slightly better achievement of fiber alignment through the convergent channel due to the superposition of such a shear flow on an elongation flow field. However, the existence of a stagnation region is not desirable for thermally unstable systems such as thermoset resins and thermally degradable thermoplastics.

iv) Fiber orientation: Rotational behavior of a specific fiber in flowing suspensions is determined by the instantaneous orientation of the fiber, the velocity gradients around the fiber center and the interaction of the fiber with surrounding fibers, resin and wall.[9,11] While plug flow is observed in low viscosity resins, the alignment of fibers in the flow direction occurs exclusively in convergent channels, where an elongational flow field exists. At higher resin viscosities there is a shear flow in the suspension near the channel wall due to the drag force induced by the wall. Because of the velocity gradient in the suspension near the wall, the fiber orientation varies not only in convergent but also in uniform channels. Particularly in convergent channels, the superposition of such a shear flow on an elongational flow field near the wall results in a slightly better fiber alignment after a convergent flow with high viscosity resins. Figure 4

shows the change in fiber orientation distribution through a 60 degree convergent channel observed in 40 vol. % suspensions as a function of flow rate and resin viscosity. Cumulative distributions of the orientation angle, ϕ_z', between the fiber axis and streamline projected onto the x-y plane, measured in the top and bottom uniform channels, are compared in Figure 4. In the figure, a 45 degree line represents the random orientation of fibers and the increase in slope indicates the increase in fiber alignment in the flow direction. The fiber alignment achieved through a 60 degree convergent channel flow shows a slight increase with increasing resin viscosity up to 1,000 poises and a sharper increase above this viscosity. However, the fiber alignment achieved through a convergent channel connecting two uniform channels is affected by the initial distribution of fiber orientations in the top channel, the change in the orientation distribution of fibers through the convergent flow and the reorientation of fibers at the exit of the convergent channel.

In a previous paper,[9] it was pointed out that the orientation angle, ϕ_z', changes with the axial position in a convergent channel as follows:

$$\tan\phi_z' = \tan\phi_{zo}' \cdot (x/x_o)^{\lambda}$$

where ϕ_z' and ϕ_{zo}' are the respective angles at the axial distances, x and x_o, from the vertex of the channel. The parameter λ is a function of the aspect ratio of the fiber and the interaction of the fiber with surrounding fibers, resin and wall.[11] The λ value observed in concentrated suspensions[9] has a broad distribution,

ranging from positive to negative values. This indicates the complexity of the fiber - fiber interaction in concentrated suspensions. When one assumes that all of the fibers oriented within the angle, ϕ'_{zo}, in the top channel are oriented within the angle, ϕ'_z, in the bottom channel, an "efficiency" parameter, λ', of the convergent channel which takes into account the reorientation of fibers at the entrance and exit of the convergent channel, can be calculated from the fiber orientation distributions in the top and bottom channels. The broken curves in Figure 4 are calculated from the initial orientation distribution (the solid curves) in the top channel and the mean value, $\bar{\lambda}'$. The mean value, $\bar{\lambda}'$, was obtained by calculating the value, λ', from

$$\tan\phi'_z = \tan\phi'_{zo} \cdot (1/4)^{\lambda'}$$

at every ten degrees of the angle, ϕ'_{zo}, and averaging the values.

Figure 5 shows the effect of resin viscosity on the mean value, $\bar{\lambda}'$, observed for a 60 degree convergent channel. The $\bar{\lambda}'$ value increases slightly with increasing resin viscosity. The effect of flow rate on the $\bar{\lambda}'$ value is negligible. The dependence of the $\bar{\lambda}'$ value on fiber concentration observed in a 444 poise resin is compared with that previously observed[9] for a 55 poise resin (the broken curve) in Figure 6. The minimum and maximum observed around 10 and 40 vol. %, respectively, in the low viscosity resin disappear in the medium viscosity resin.

The $\bar{\lambda}'$ value slightly increases with increasing fiber concentration in the latter case. The relation between convergence angle and the $\bar{\lambda}'$ value observed in the above medium viscosity resin is shown in Figure 7. The broken curve represents the fiber behavior observed in a low viscosity resin.[9] The $\bar{\lambda}'$ value decreases with increasing the convergence angle in both the cases. The effect of resin viscosity on the convergence angle dependence of fiber alignment is minor in the viscosity range, as shown in the figure.

v) <u>Flow properties</u>: The flow resistance, defined here as the load required to produce a constant flow rate through a convergent channel followed by a uniform channel, is a function of resin viscosity, fiber orientation distribution, velocity profiles and relative velocity of fibers to the resin. The flow resistance fluctuates with time,[12] if the flow in the convergent channel is unstable due to the changes in fiber orientation distribution, velocity profile and resin - fiber separation. Such instabilities in the flow resistance, observed with 40 vol. % suspensions subjected to flow through a 34.8 degree convergent channel, are shown in Figure 8. The channel exit has the dimensions of 1/8" x 1/8", and is easily blocked with 1/8" long fibers when the resin viscosity is as low as 55 poises.[9] This is always demonstrated by a sudden increase in the flow resistance. To achieve a more steady flow in low

viscosity resins, a wider exit of 1/8" x 1/4" is used in the present experimental study. For a comparison of flow resistances observed in various suspensions, the first plateau in load is used, since this represents independence of load with the plunger displacement in the top channel. This is illustrated by the dotted lines in Figure 8.

The dependence of flow resistance on resin viscosity observed for 40 vol. % suspensions is shown in Figure 9. These suspensions were subjected to flow through a 60 degree convergent channel; the solid curves are the best fit to the experimental data. The increase in the flow resistance of suspensions with increasing resin viscosity is minor when the resin viscosity is less than 1,000 poises. Above this viscosity a sharp increase is observed, as shown in Figure 9. It is noted here that the resin mixtures (i) are unstable and that the resin viscosity increases 30 to 100 % 48 hours after preparation. The other resin mixtures (ii) are very stable, and the resin viscosity does not change after several months. When the resin is unstable, the flow resistance of the suspension seems to be lower than that for stable resins, presumably due to the uneven viscosity values outside and inside the fiber granules prepared prior to the flow experiments.

The flow resistance of suspensions increases with increasing fiber concentration and convergence angle, as illustrated in Figures 10 and 11. The dependence of flow resistance on fiber concentration, observed for suspensions with a medium viscosity resin (444 poises) is shown in Figure 10. In this resin, elimination of trapped air bubbles is most difficult at 10 vol. %, as previously observed in a low viscosity resin.[9] The increase in flow resistance of 5 and 10 vol. % suspensions with decreasing flow rate may be attributed to the momentary blockade of the channel exit with fibers. The dependence of flow resistance on convergence angle, observed for 40 vol. % suspensions with a 444 poise resin, is shown in Figure 11; the solid curves are the best fit to the experimental data. Flow resistance increases with increasing convergence angle, in general, but becomes almost independent of convergence angle when the angles are larger than 120 degrees. As pointed out before, the stagnation region increases and the actual converging flow of suspensions has smaller convergence angles when the convergence angle of channel is larger than 120 degrees.

CONCLUSIONS

The effect of resin viscosity on the orientation, distribution and deformation of short fibers in concentrated suspensions flowing through rectangular channels and the flow properties of the suspensions can be summarized as follows:

i) When resin viscosity is lower than 800 poises, plug flow is observed in 30 to 50 vol. % suspensions subjected to flow through uniform and convergent channels. The alignment of fibers in the flow direction occurs exclusively in convergent channels. When resin viscosity is higher than 1,000 poises, however, velocity profiles observed in uniform and convergent channels are similar to those for Newtonian fluids but blunted near the channel center. The alignment of fibers in the flow direction occurs in both convergent and uniform channels.

ii) Resin - fiber separation decreases with increasing resin viscosity.

iii) The fiber alignment achieved through a convergent channel flow increases as resin viscosity increases.

iv) More buckling and debundling of fibers are observed as resin viscosity increases.

v) The flow resistance of 40 vol. % suspensions extruded through a 60 degree convergent channel slightly increases with increasing resin viscosity up to 1,000 poises and sharply increases above this resin viscosity.

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Table I. The relative velocity, V_{fiber}/V_{mean}, of fibers to the mean velocity in 40% suspensions subjected to flow through convergent channels.

Resin viscosity: (poises)	54.8		444		700
Flow rate: (in³/min.)	0.250	0.625	0.250	0.625	0.250
In a 20° channel:	blocked.	0.99 ± 0.12	0.85 ± 0.12	0.95 ± 0.12	0.97 ± 0.09
In a 30° channel:	0.79 ± 0.37	0.85 ± 0.22	0.79 ± 0.13	0.93 ± 0.15	1.03 ± 0.12
In a 60° channel:	0.92 ± 0.12	0.79 ± 0.21	0.86 ± 0.09	0.89 ± 0.15	0.91 ± 0.18

Table II. The relative velocity, V_{fiber}/V_{mean}, of fibers to the mean velocity in concentrated suspensions flowing through uniform channels.

Resin viscosity: (poises)	54.8		444	
Flow rate: (in³/min.)	0.250	0.625	0.250	0.625
a) In the top, 1/8" x 1", channel:				
In 30% suspension:	0.88 ± 0.09	-	0.87 ± 0.04	0.84 ± 0.09
In 40% suspension:	0.89 ± 0.05	0.89 ± 0.16	0.92 ± 0.05	0.91 ± 0.06
In 50% suspension:	0.92 ± 0.05	-	0.85 ± 0.04	0.89 ± 0.05
b) In the bottom, 1/8" x 1/4", channel:				
In 30% suspension:	0.78 ± 0.03	-	curve	curve
In 40% suspension:	0.93 ± 0.05	0.90 ± 0.13	0.95 ± 0.12	1.00 ± 0.07
In 50% suspension:	0.83 ± 0.14	-	0.84 ± 0.17	0.98 ± 0.06

LIST OF FIGURES

Figure 5. The effect of resin viscosity on the mean value $\bar{\lambda}'$ observed in 40 vol. % suspensions flowing through a 60° convergent channel at the flow rates of 0.250 in.^{3}/min. (closed circles).

Figure 6. The effect of fiber concentration on the mean value $\bar{\lambda}'$ in suspensions with a 444 poise resin which flow through a 60° convergent channel at the flow rates of 0.250 in.^{3}/min. (open circles) and 0.625 in.^{3}/min (closed circles). The solid curve is the best fit to the present data and the broken curve represents the relation observed in the suspensions with a 55 poise resin.[9]

Figure 7. The effect of convergence angle on the mean value $\bar{\lambda}'$ observed in 40 vol. % suspensions with a 444 poise resin which are subjected to flow through the convergent channels at the flow rates of 0.250 in.^{3}/min. (open circles) and 0.625 in.^{3}/min. (closed circles). The solid curve is the best fit to the present data and the broken curve represents the relation observed in suspensions with a 55 poise resin.

Figure 8. Fluctuation of the load required to maintain a constant flow rate, 0.250 in.^{3}/min., through a 34.8° convergent channel. Fiber concentration is 40 vol.% in the suspensions. The channel exit has the dimensions of 1/8" x 1/8".

Figure 9. The effect of resin viscosity on the flow resistance observed for 40 vol. % suspensions flowing through a 60° convergent channel: O and Δ, for the resin mixtures (ii) and ●, for the resin mixtures (i).

Figure 10. The effect of fiber concentration on the flow resistance observed for the suspensions with a 444 poise resin which are subjected to flow through a 60° convergent channel.

Figure 11. The effect of convergence angle on the flow resistance of 40 vol. % suspensions with a 444 poise resin: the solid curves are the best fit to the present data and the broken curves represent the relations previously observed for the suspensions with a 55 poise resin.[9]

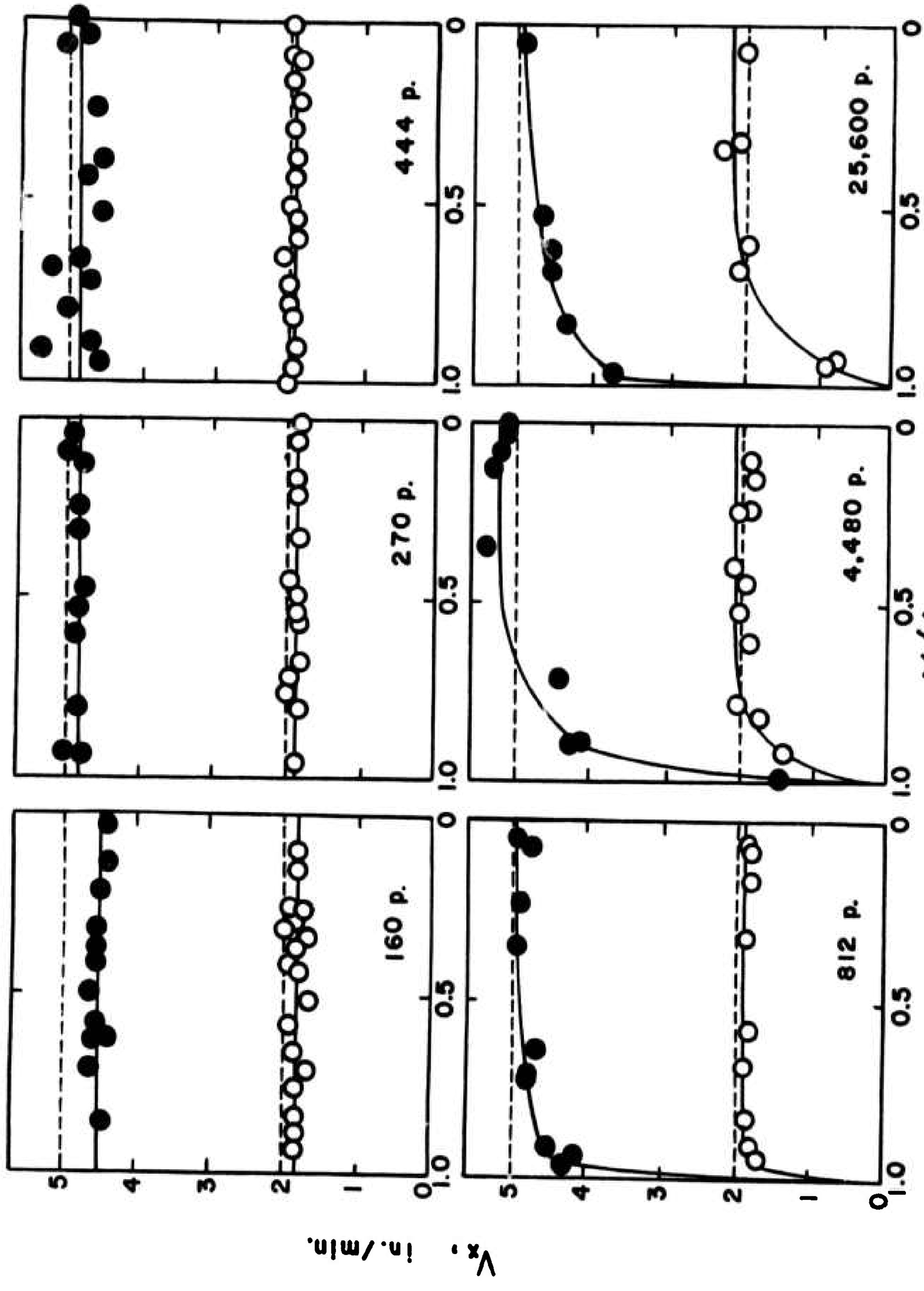

Figure 1

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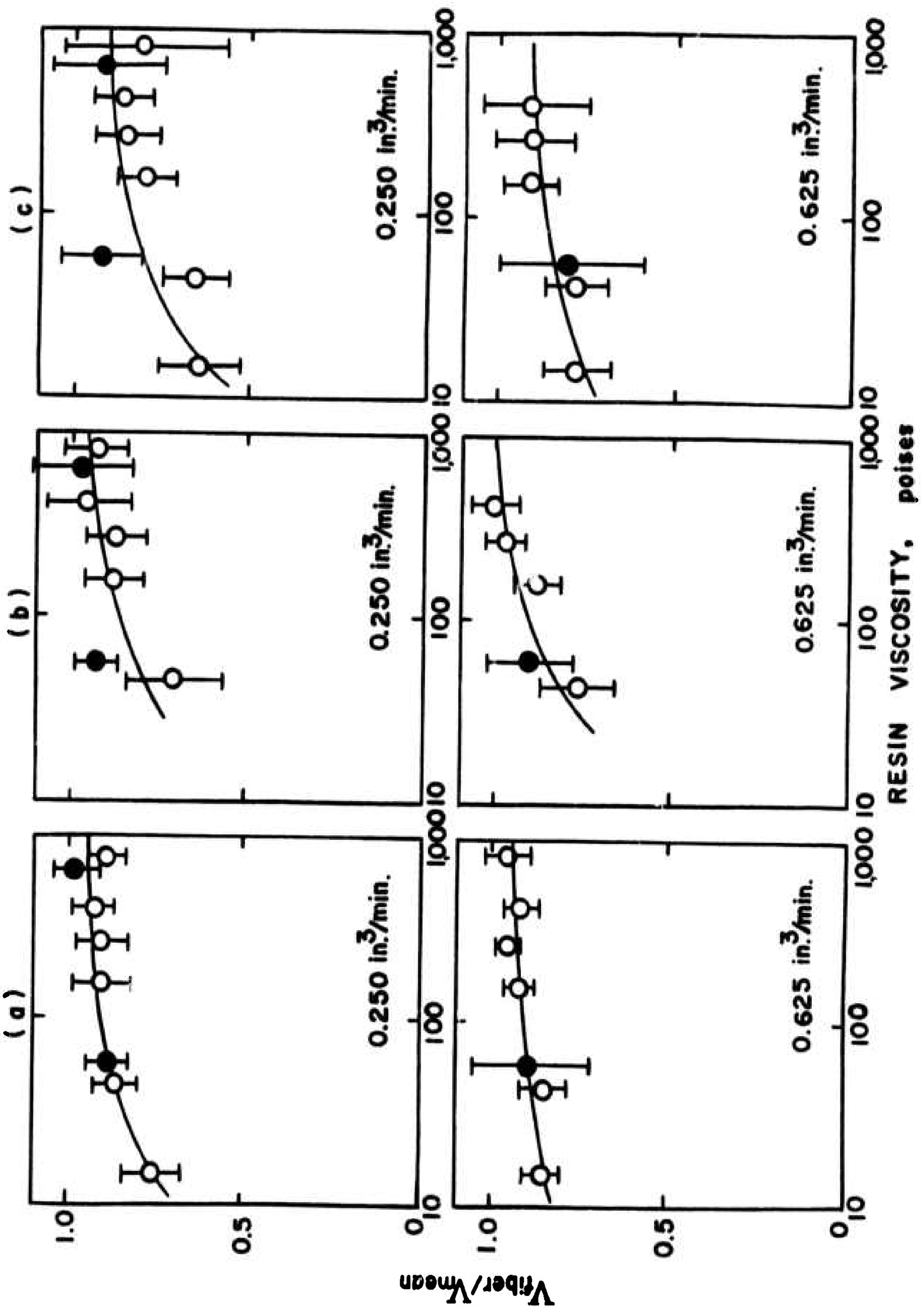
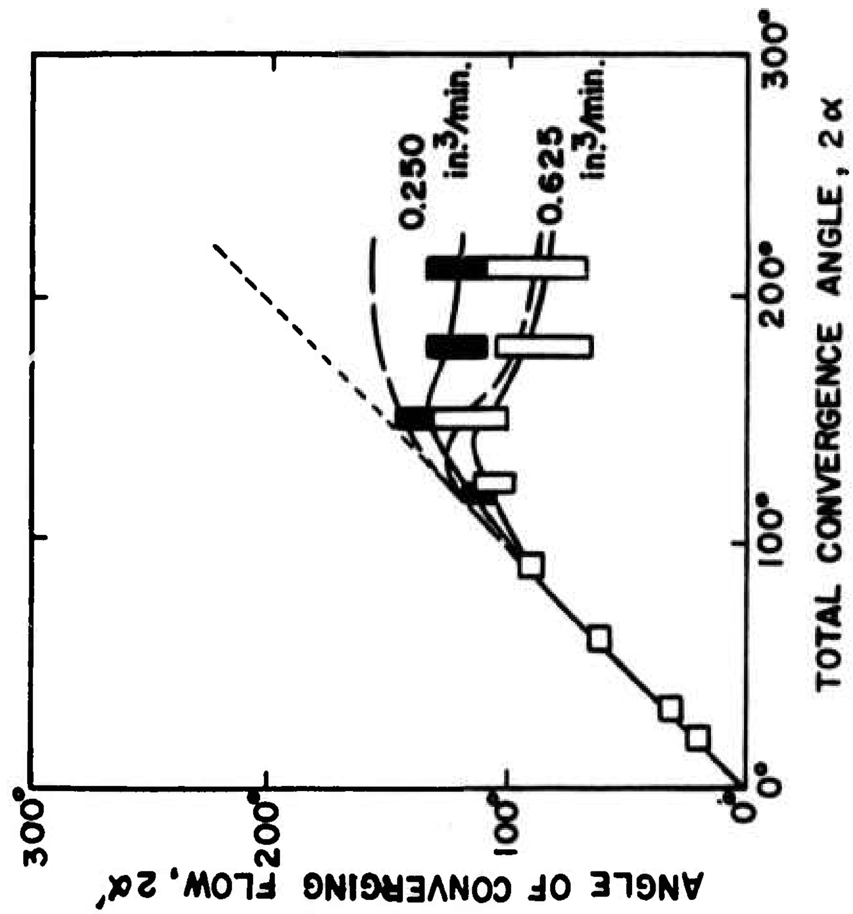


(a)
(b)
(c)
0.250 in.³/min.
0.625 in.³/min.
RESIN VISCOSITY, poises
Vfiber/Vmean
1.0
0.5
0
10
100
1,000

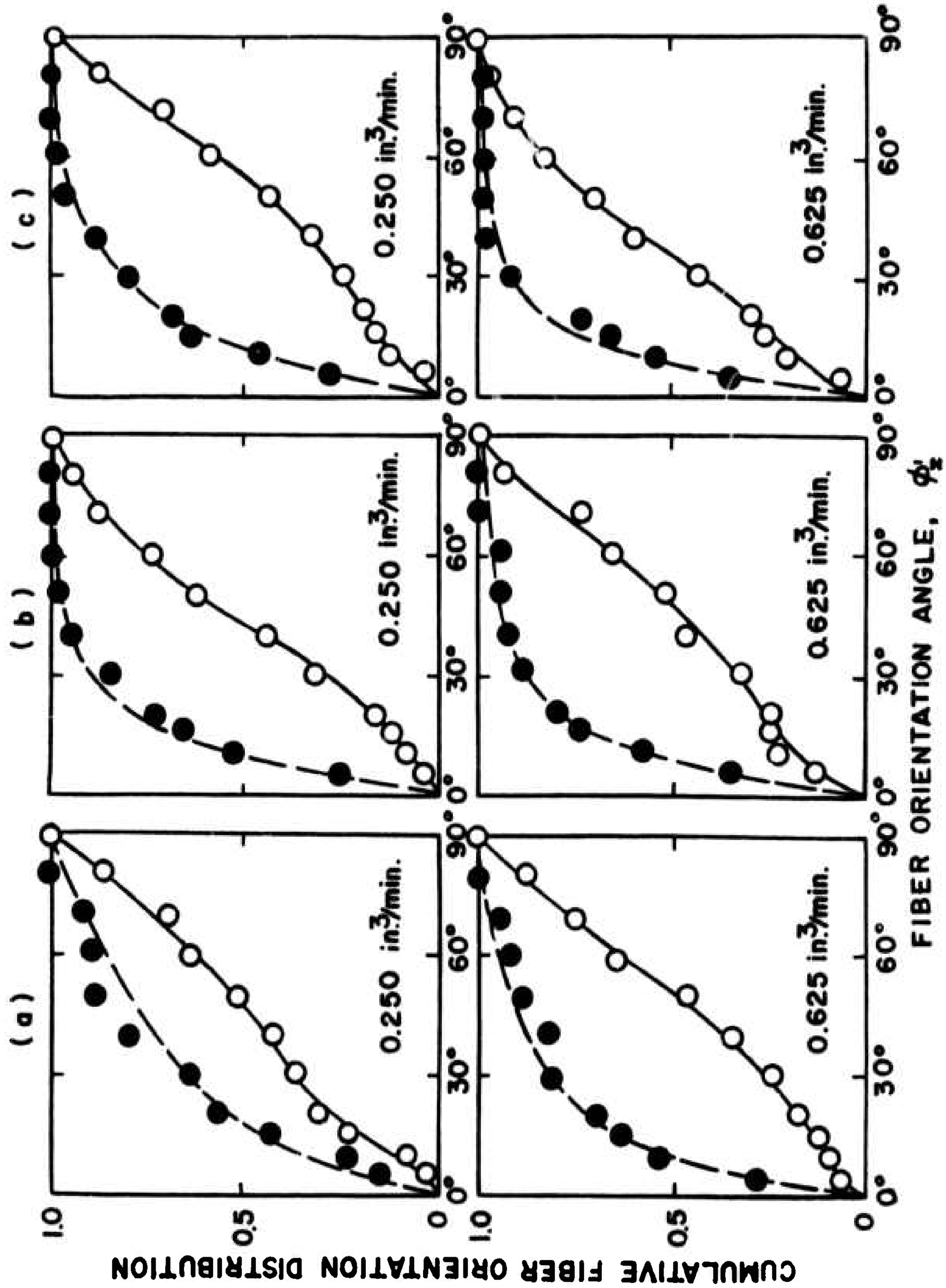
Figure 2



0.250 in.3/min.
0.625 in.3/min.
0°
100°
200°
300°
TOTAL CONVERGENCE ANGLE, 2α
ANGLE OF CONVERGING FLOW, 2α'

Figure 3

26



(a)
(b)
(c)
0.250 in.^{3}/min.
0.625 in.^{3}/min.
FIBER ORIENTATION ANGLE, ϕ_z'
CUMULATIVE FIBER ORIENTATION DISTRIBUTION

Figure 4

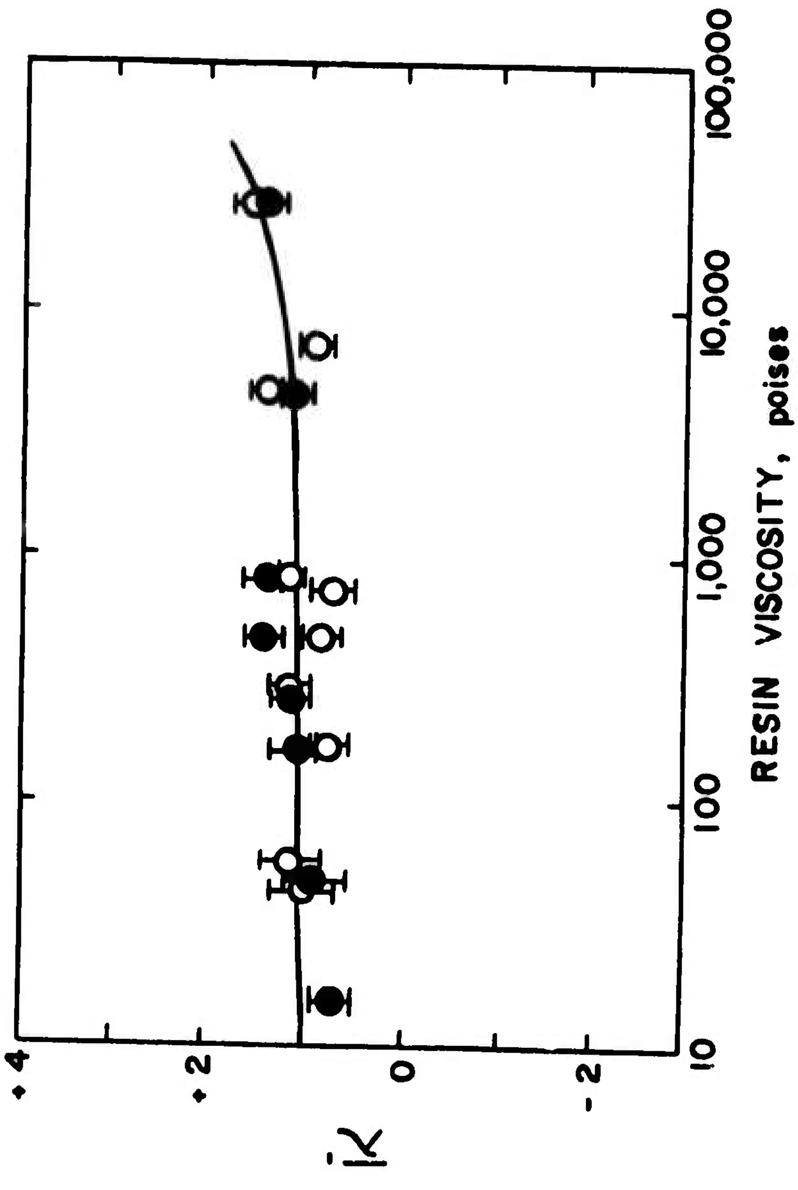


Figure 5

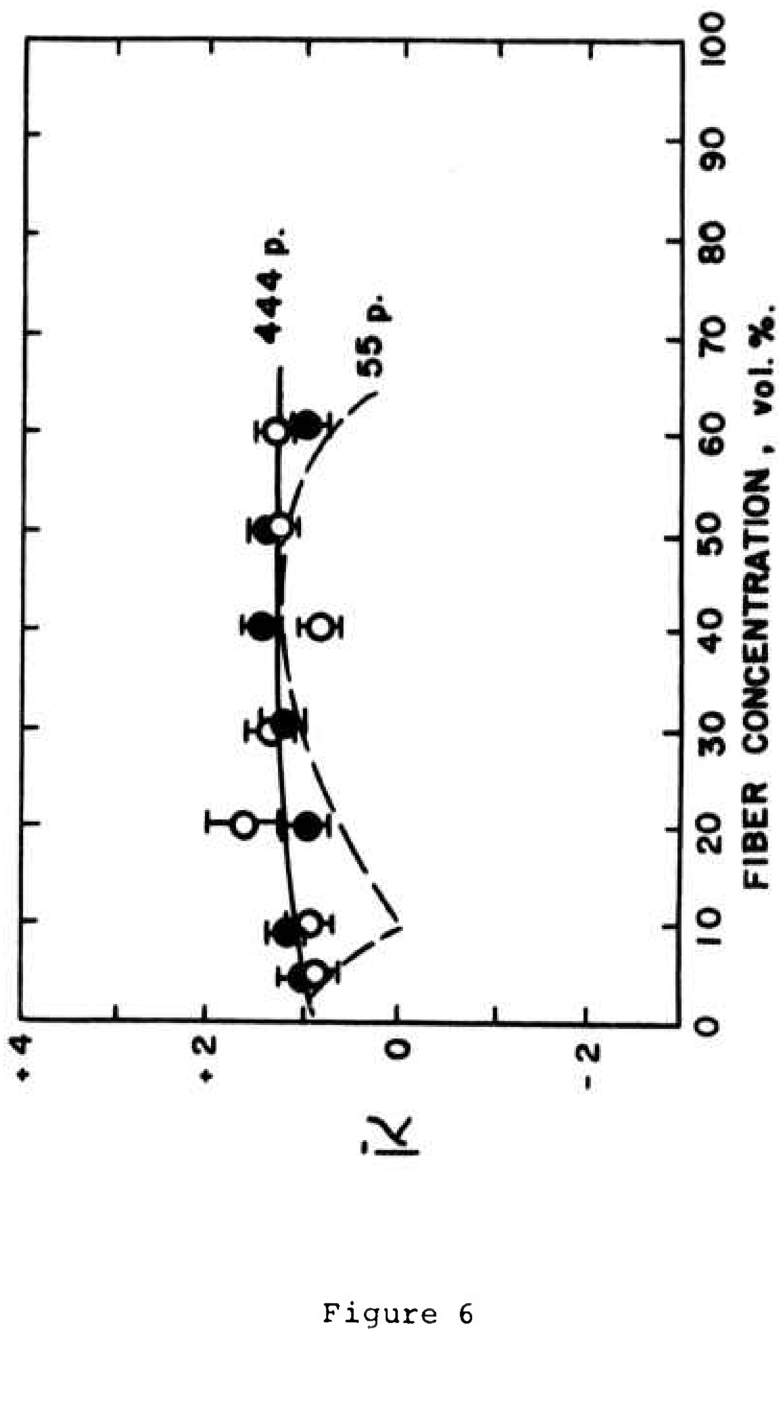

444 p.
55 p.
FIBER CONCENTRATION , vol. %.
0
10
20
30
40
50
60
70
80
90
100
+4
+2
0
-2

Figure 6

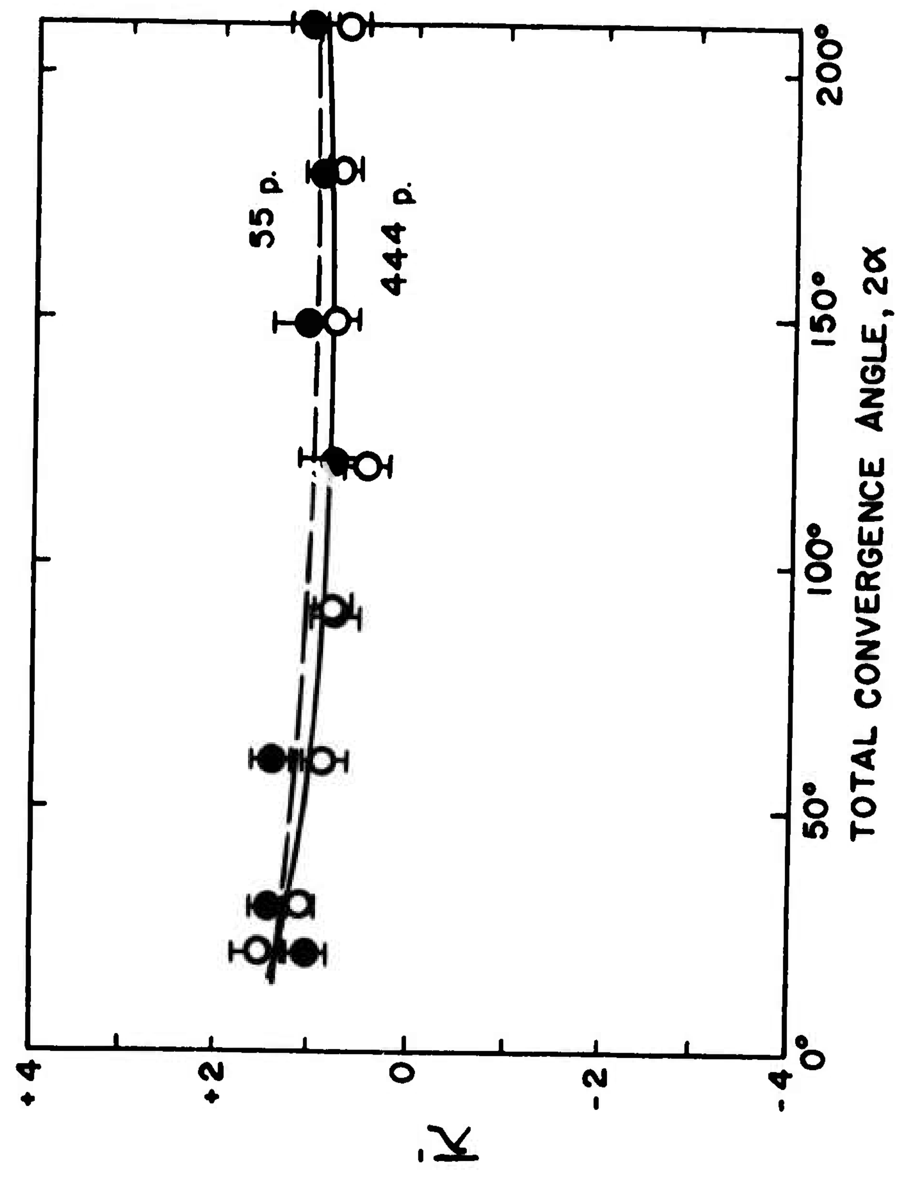


Figure 7

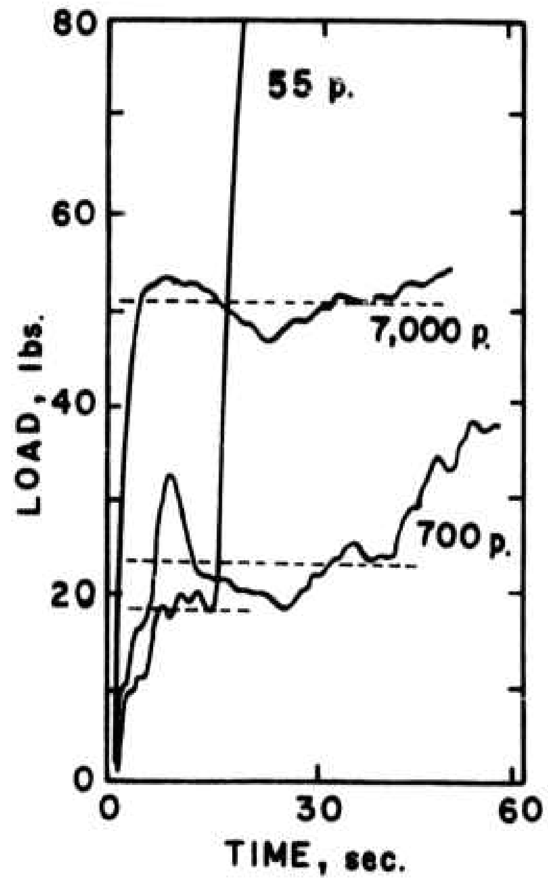


Figure 8

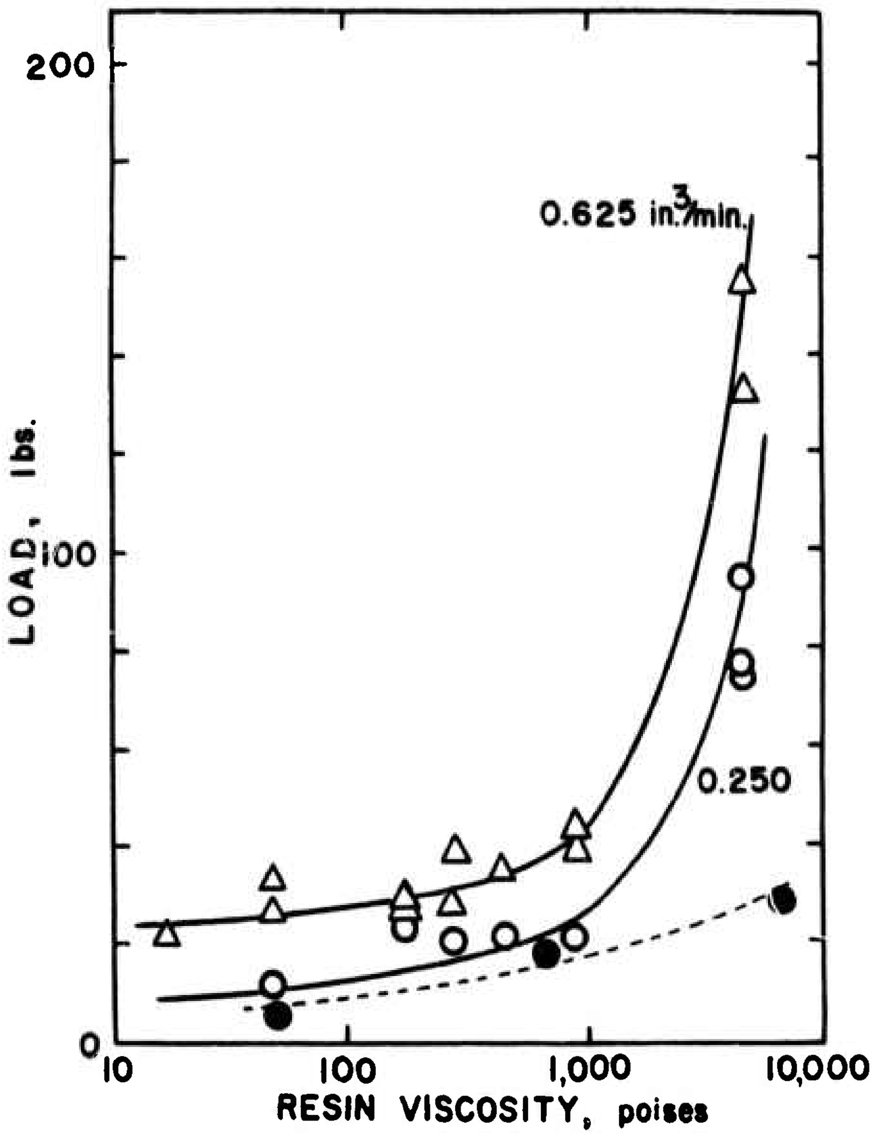

200
100
0
LOAD, lbs.
0.625 in.³/min.
0.250
10
100
1,000
10,000
RESIN VISCOSITY, poises

Figure 9

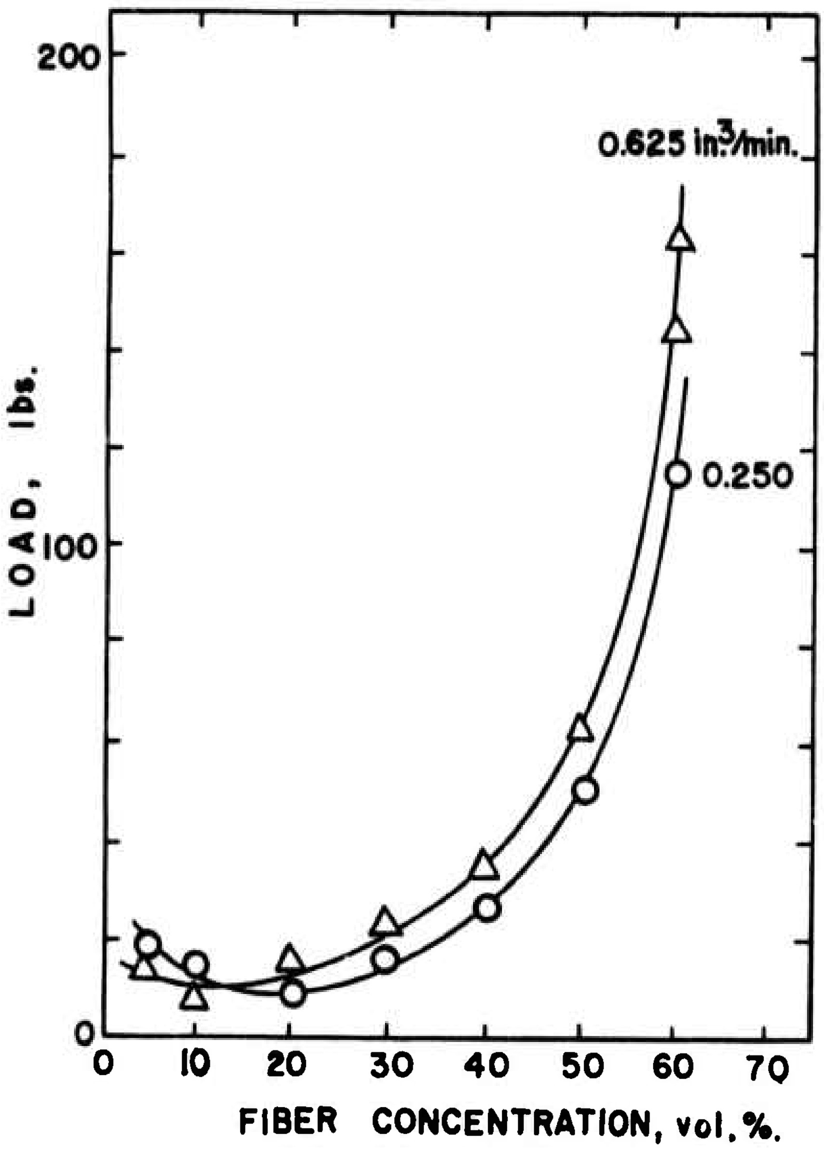

200
100
0
0.625 in.³/min.
0.250
LOAD, lbs.
0
10
20
30
40
50
60
70
FIBER CONCENTRATION, vol. %.

Figure 10

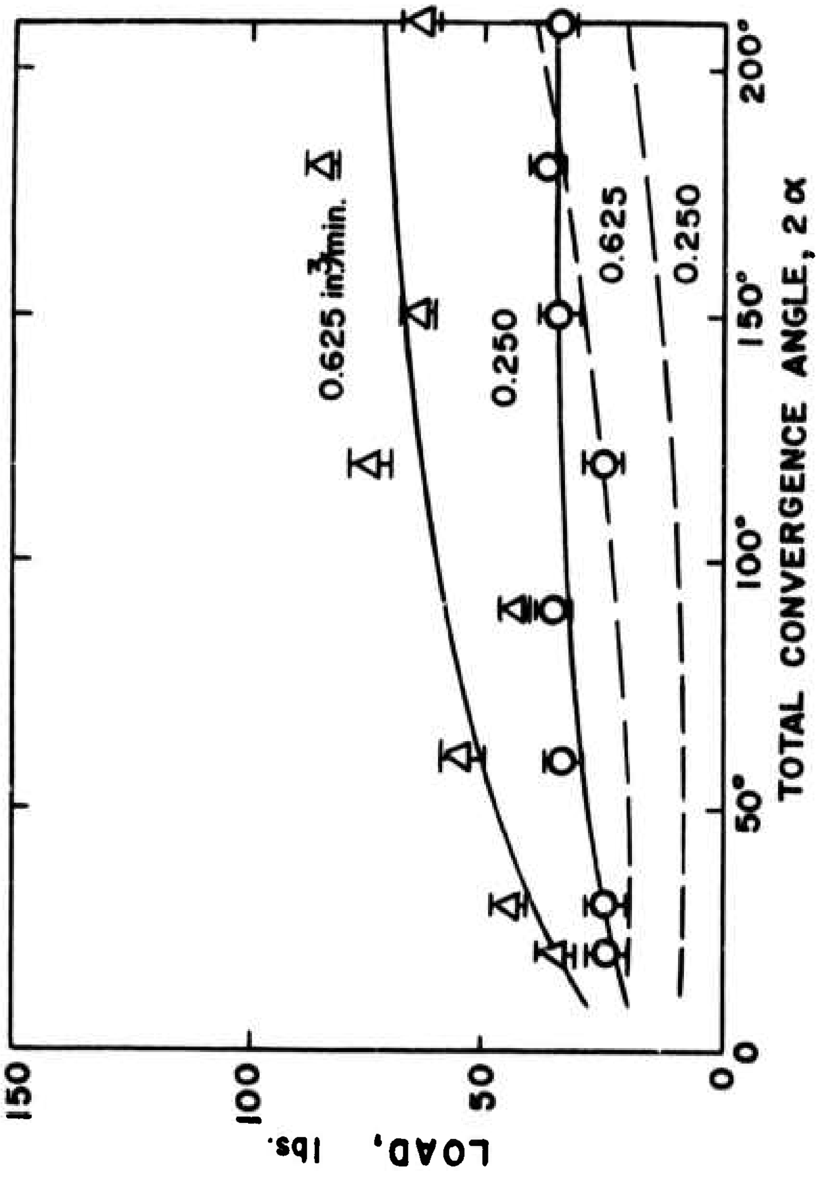

LOAD, lbs.
0
50
100
150
TOTAL CONVERGENCE ANGLE, 2 α
0
50°
100°
150°
200°
0.625 in³/min.
0.250
0.625
0.250

Figure 11